

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
14 February 2002 (14.02.2002)

PCT

(10) International Publication Number
WO 02/11691 A1

(51) International Patent Classification⁷: **A61K 7/32**

(21) International Application Number: PCT/GB01/02882

(22) International Filing Date: 29 June 2001 (29.06.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
0019230.2 4 August 2000 (04.08.2000) GB

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(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

- with international search report
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: ANTIPERSPIRANT FORMULATIONS

(57) Abstract: Anhydrous antiperspirant formulations in which a particulate antiperspirant active is suspended in a carrier fluid can result in significant visible deposits when applied to skin or when transferred onto clothing. Anhydrous antiperspirant suspension sticks which exhibit very low visible deposits are obtainable by employing a carrier fluid in which at least 45% and preferably at least 60% of its weight comprises a mixture of a hydrocarbon oil with an oxygen-containing emollient oil, the mixture having a refractive index of at least 1.46, in conjunction with structurant comprises a wax or a non-polymeric fibre-forming gellant. Preferred oxygen-containing emollient oils include alkyl benzoates. The waxes can even comprise fatty alcohols.



WO 02/11691 A1

- 1 -

Antiperspirant Formulations

The present application relates to antiperspirant formulations, and in particular to antiperspirant stick formulations.

Background and Prior Art

10 Topically applied antiperspirant compositions are in widespread use throughout much of the world, in order to enable their users to avoid or minimise visible wet patches on their skin, especially in axillary regions. Antiperspirant formulations containing astringent metal salts such as aluminium or zirconium salts commonly act also as deodorants. Antiperspirant formulations have been produced or proposed for use in several physical forms, such as solids, gels, soft solids, creams, lotions and particulate mixtures and these forms can be applied using a range of different dispensers, including aerosol, roll-on, pump spray, sticks, and barrel dispensers, in accordance with the individual preferences of consumers. In some parts of the world, solid formulations in stick form are especially popular, the stick usually being dispensed from a barrel provided with means to expose the stick above the barrel.

Herein, the term stick indicates a bar of solid material which retains its integrity whilst being applied, i.e. a firm stick and which is commonly, though not exclusively, housed within a dispensing container which hitherto is

- 2 -

conveniently in the shape of a barrel. When a portion of a firm stick is drawn across the skin surface, a film of the stick composition is transferred onto the skin surface. Although the stick has the appearance of a solid article,
5 the material forming the stick usually comprises a structured liquid phase such that a film of the material is readily transferred onto another surface such as axillary skin upon contact under pressure.

10 There are typically three classes of firm antiperspirant sticks, namely suspension sticks, emulsion sticks and solution sticks. Suspension sticks contain a particulate antiperspirant active material suspended in a structured carrier. Emulsion sticks normally comprise an emulsion of
15 an oil phase and a hydrophilic phase containing the antiperspirant active in solution, the continuous phase being structured. In some emulsion sticks, the continuous phase is an oil phase. In solution sticks, the antiperspirant is typically dissolved in the liquid carrier
20 phase which is structured. The liquid phase can comprise water and/or a water-miscible organic solvent. The three categories can be applied to sticks of both firm and soft solids compositions.

25 Conventionally, many suspension sticks have been structured using naturally-occurring or synthetic waxes, of which typical examples include stearyl alcohol, hydrocarbon waxes, waxes of plant or animal origin or their synthetic analogues or derivatives or silicone waxes. Waxes are widely
30 available, and by suitable selection of the waxes themselves and their concentrations in the formulation can effectively

- 3 -

obtain either a soft solid or a firm solid. Thus for example, wax-structured sticks are described in an article in Cosmetics and Toiletries, 1990, vol. 105, p75-78.

However, many conventional fatty alcohol or other wax structured sticks tend to leave visible white deposits on application to human skin, and likewise, visible deposits can also be transferred onto clothing by physical contact with the skin. Such visible deposits are disliked by a significant, and in some countries growing, proportion of

consumers of antiperspirants, be they on the skin or on clothing. Accordingly, the antiperspirant industry, including the instant inventors, is continuing devoting considerable time and resources to finding means to ameliorate or overcome customer perceived whiteness deposits. In countries where both traditional and low whitening antiperspirant stick products are available, sales of the low whitening formulations have grown relative to traditional formulations.

Patents and patent documents relevant to this field of invention include:

USP 3,255,082;	Barton et al;
USP 3,986,203;	Spitzer et al;
USP 4,083,956,	Shelton;
EP-A-0,028,853,	Beckmeyer et al;
USP 4,425,328,	Nabial et al;
USP 4,265,878,	Keil;
USP 4,229,432,	Geria;
USP 4,724,139,	Palinczar
USP 4,985,238,	Tanner et;

- 4 -

USP 5,486,347, Callaghan et al and
USP 6,068,518, Bianchi et al.

Antiperspirant formulations remain on the skin for
5 considerable periods of time after application, for example
many hours before the axilla or other part of the body to
which the antiperspirant has been applied is washed. In
that respect they differ significantly from many other
personal care formulations such as washing or cleansing
10 formulations which are applied and almost immediately
removed. Accordingly, all non-volatile components present
in the antiperspirant formulations remain on the skin,
including not only non-volatile anti-perspirant actives, but
also non-volatile components of any carrier fluid which is
15 employed. Such non-volatile components not only remain on
the skin, but likewise remain available for transfer onto
clothing which comes into contact with the skin. Carrier
liquid components can assist in the transfer of solids
suspended therein.

20 Since their introduction, volatile silicone fluids have been
widely accepted within the antiperspirants industry as
carrier fluids for antiperspirant solids, and commonly are
either thickened to form a cream or structured to form a
25 solid in order to suspend particulate antiperspirant
materials. Volatile silicone oils have proven to be very
popular, on account of their combination of beneficial
properties, so that in many or most of the common commercial
anhydrous suspension stick formulations, such oils
30 constitute the principal proportion of the carrier fluid.
One or more other oils, such as non-volatile silicone oils

- 5 -

or non-volatile non-silicone emollient oils are often included or proposed for inclusion in patent specifications in minor amounts in firm stick suspended antiperspirant formulations, and indeed together commonly constitute no
5 more than a minor fraction of the carrier fluid.

Although volatile silicone oils enable the antiperspirant formulations to exhibit many desirable properties, there are two consequences of employing them as the principal
10 component of the carrier fluid, namely that such antiperspirant formulations tend to exhibit comparatively high visible whiteness when they are topically applied and also the visible whiteness of such formulations tends to increase further with the passage of time. Without being
15 bound to any theory, it is believed that increased visible whiteness arises from evaporation of the volatile silicones, thereby exposing those formulation components such as particulate antiperspirant actives and/or structurant which contribute to visible whiteness. These observations are of
20 relevance, because users look carefully at where they are applying antiperspirant formulations, and they often remain on the skin for extended periods of time.

However, it is no simple matter to reformulate
25 antiperspirant formulations with alternative oils. Whilst many oils have been accepted within the industry as minor components of a volatile-silicone-based carrier fluid, the very properties which have rendered them suitable to be considered suitable as emollients have been thought to
30 render them unsuitable as the principal carrier oil. These

- 6 -

properties include a propensity to oiliness or greasiness in the resultant product, when present in a large proportion.

A number of attempts have been made already to address the problems of selecting carrier fluids for antiperspirant formulations. Several patent specifications offer lists of emollient oils for incorporation in carrier fluids, in some instances for creams, such as in EP-A-388110 and WO-A-98/51272 and in other instances for sticks, such as US-A-5833964. These specifications classify as similar, materials which the investigation resulting in the present invention show to be significantly different. Accordingly, although such specifications assert that they provide formulation exhibiting low visible deposits, this assertion is often judged against those sticks which were commercially available when the applications were sought and in practice, there often remains considerable room for improvement and do not provide adequate teaching to the skilled formulator to enable that person to choose effectively. Other patent specifications have disclosed the use of certain specified types of hydrocarbons, such as poly alpha olefins in US-A-4919934 and EP-A-804921, in respectively sticks or creams. In view of the feel associated with hydrocarbons, it remains a desideratum to provide formulations which have a lower non-volatile hydrocarbon content.

Object of the invention

It is an object of the present invention to ameliorate or overcome one or more of the disadvantages of suspension antiperspirant formulations disclosed hereinabove.

- 7 -

It is a further object of at least some embodiments of the present invention to provide a suspension formulation employing a carrier having a non-volatile oil as principal carrier.

5

Brief Description of the Invention

According to the present invention there is provided an
10 anhydrous antiperspirant formulation comprising a
particulate antiperspirant suspended in a water-immiscible
carrier fluid which is structured by an effective amount of
a structurant, characterised in that at least 45% by weight
of said carrier fluid comprises a mixture of at least two
15 emollient oils comprising a hydrocarbon oil and an oxygen-
containing emollient oil, preferably in a weight ratio of
from 7:3 to 1:9, said mixture having a refractive index of
at least 1.46 and said structurant comprises a wax or a non-
polymeric fibre-forming gellant.

20

Herein, and in order for said mixture of emollient oils to
achieve a refractive index of at least 1.46, at least one of
the oils needs have a higher refractive index, which is
often at least 1.465. This is commonly a suitably chosen
25 oxygen-containing emollient. An emollient oil which has a
refractive index of at least 1.465 is often abbreviated
herein to HR emollient, and in many instances reference is
also made to the class of oil.

30 The refractive index of the mixture of emollient oils can be
obtained by measurement (herein always at 22°C unless

- 8 -

otherwise specified) using a refractometer. An approximation can be obtained by calculating a weighted average of the refractive indices of the individual components of the mixture.

5

By employing a mixture of emollient oils having the designated refractive index and comprising a hydrocarbon oil and an oxygen-containing emollient oil such as in the stated weight ratio range and in the stated proportion of the carrier fluid, it is possible to obtain sticks exhibiting an excellent combination of attributes, ameliorating or avoiding undesirable attributes associated with formulations that employ predominantly volatile silicones or liquid hydrocarbons alone as the carrier fluid.

10

By the choice of such a combination of such a carrier fluid components and structurant, it has been possible to obtain antiperspirant sticks showing excellently low visible deposits.

15

In a second aspect of the present invention there is provided a process for the production of an antiperspirant stick comprising the steps of:

20

i. incorporating into a liquid carrier a structurant at an effective concentration and in an amount sufficient to structure the carrier to render it solid at 20°C;

ii. rendering the structurant-containing mixture or one or more of its constituents mobile at an elevated temperature;

25

iii. mixing the liquid carrier with an antiperspirant

30

active to form an antiperspirant-containing mixture, steps 2

- 9 -

and 3 being conducted either before, after or simultaneously with step 1;

iv. introducing the mobile mixture into moulding means and

v. cooling or permitting the mobile mixture to cool to a

5 temperature at which it is structured, characterised in that

at least 45% by weight of said carrier fluid comprises a

mixture of at least two emollient oils comprising a

hydrocarbon oil and an oxygen-containing emollient oil,

preferably in a weight ratio of from 7:3 to 1:9, said

10 mixture having a refractive index of at least 1.46

and said structurant comprises a wax or a non-polymeric

fibre-forming gellant.

In a third aspect, there is provided a method of reducing or

15 controlling axillary sweating comprising applying topically

to skin an anhydrous antiperspirant formulation comprising a

particulate antiperspirant suspended in a water-immiscible

carrier fluid which is structured by an effective amount of

a structurant, characterised in that at least 45% by weight

20 of said carrier fluid comprises a mixture of at least two

emollient oils comprising a hydrocarbon oil and an oxygen-

containing emollient oil, preferably in a weight ratio of

from 7:3 to 1:9, said mixture having a refractive index of

at least 1.46 and said structurant comprises a wax or a non-

25 polymeric fibre-forming gellant.

In a fourth aspect of the present invention there is

provided an antiperspirant product comprising an

antiperspirant formulation in the form of a stick disposed

30 within a packaging which enables a portion of the stick to

be exposed for topical application to skin characterised in

- 10 -

that the formulation comprises a particulate antiperspirant
suspended in a water-immiscible carrier fluid which is
structured by an effective amount of a structurant,
characterised in that at least 45% by weight of said carrier
5 fluid comprises a mixture of at least two emollient oils
comprising a hydrocarbon oil and an oxygen-containing
emollient oil, preferably in a weight ratio of from 7:3 to
1:9, said mixture having a refractive index of at least 1.46
and said structurant comprises a wax or a non-polymeric
10 fibre-forming gellant.

Detailed Description of the Invention and Preferred
Embodiments

15

The present invention provides antiperspirant sticks
structured with wax and/or non-polymeric fibre-forming
gellant in which a particulate antiperspirant active is
suspended in a carrier fluid comprising a substantial
20 fraction of mixture of emollient oils, said mixture having a
refractive index of at least 1.46.

The choice of such a mixture of emollient oils in a high
proportion of the carrier fluid is of importance in the
25 present invention. In essence, the mixture of the emollient
oils satisfies three criteria. First, it is a mixture of
anhydrous water-immiscible oils, each of which is fluid at
ambient temperature, such as 20°C. Secondly, at least one
oil in the mixture has a relatively high refractive index,
30 such as above 1.465 and thirdly within the mixture, the

- 11 -

oxygen-containing emollient oil contributes a substantial fraction.

By the choice of such a mixture of HR emollient oils, and by
5 employing it in a high proportion of the carrier fluid, it
is possible to obtain sticks with a wide choice of
structurant, but which leave no higher than very low visible
white deposits on skin or clothing. The ability to have a
wide choice of structurant is of considerable benefit to
10 antiperspirant manufacturers in that it means that they are
not restricted to a narrow range of materials. The
resultant low or very low visible deposits, even when
employing waxes which have acquired a reputation for leaving
high or comparatively high visible deposits, is manifestly
15 of benefit in view of the prevailing desire expressed by
consumers for antiperspirants which do not leave unsightly
marks on clothing or on the skin surface.

Most if not virtually all of the HR oxygen-containing
20 emollients suitable for use in the present invention have a
refractive index of at least 1.465, and often at least 1.47
and most of up to 1.56. A number of especially desirable HR
oxygen-containing emollients have a refractive index in the
region of 1.47 to 1.49. They can be employed individually
25 or mixed together. Yet other desirable HR oxygen-containing
emollients have a refractive index in the region of 1.50 to
1.56, which can also be used individually or mixed together.
If desired, it is possible to employ a mixture of an HR
emollient having a refractive index of up to 1.49 with that
30 having a refractive index of at least 1.50. Mixtures of HR
emollients can be selected at weight ratios of the

- 12 -

individual emollients at the discretion of the formulation maker.

It is highly desirable to employ emollients which impart
5 little or no colour to formulations containing them.
Accordingly, it is preferred to avoid or minimise the
incorporation of alkoxy cinnamates, even if such compounds
demonstrate a refractive index of above 1.5, such as by
restricting the content of such cinnamates being cinnamate-
10 deficient by which herein we mean below 5% of the weight of
the carrier fluid, or preferably free from cinnamate by
which we mean below 1% and especially down to 0%. Such
cinnamates tend to be highly coloured, especially in the
yellow region of the spectrum, and would render
15 antiperspirant stick less acceptable or even unacceptable to
potential consumers. Likewise, there are two further
reasons for avoiding or minimising the incorporation of
such alkoxy cinnamates. It is desirable to avoid employing
or at least minimise the presence of any emollient which has
20 an intrinsically high and unpleasant odour or one which
would cause unacceptable irritation.

The proportion of the emollient mixture in the carrier fluid
is at in practice at least 45% by weight, and in many
25 desirable formulations is at least 50% or from 55% by
weight. If desired, the mixture can comprise as much as
100% of the carrier fluid. However, in practice, the
formulation also includes a number of other liquid
constituents such as fragrance or other liquid emollients,
30 so that it commonly provides no more than 99% and in a
considerable fraction of suitable formulations, up to 95% by

- 13 -

weight of the carrier fluid. In a number of formulations, the proportion of HR emollient is at least 60% by weight and more desirably at least 70% by weight and especially at least 80% by weight of the carrier fluid. In a preferred
5 range, the HR emollient comprises from 70 to 99% by weight of the carrier fluid.

The hydrocarbon oils, which can be contemplated for the non-HR oxygen-containing emollient oils comprise liquid
10 aliphatic hydrocarbons such as mineral oils or hydrogenated polyisobutene, often selected to exhibit a low viscosity. Poly alpha olefins constitute useful liquid hydrocarbons herein. Further examples of liquid hydrocarbons are polydecene and paraffins and isoparaffins of at least 10
15 carbon atoms. Although polyisobutene and polydecene are polymeric in nature, they are mobile liquids at room temperature of 20°C and do not cause thickening of other hydrophobic oils. Some mineral oils have an RI which approaches 1.465 and accordingly mixtures containing them
20 have both a hydrocarbon and an oxygen-containing emollient above the minimum average value for refractive index of 1.46.

The HR oxygen containing emollients employed in the present
25 invention contain within the oil at least one oxygen atom, which may be embedded within the backbone of the emollient or in a side chain or comprise a substituent. There are two particularly desirable classes of oxygen-containing HR emollients, namely a) alkylated or alkoxyated benzoates and
30 b) non-volatile silicone oils. In class a) the oxygen is embedded within a carbon backbone i.e. a C-O-C sub-structure

- 14 -

and within class b) within a silicon backbone, i.e. an Si-O-Si sub-structure.

Within class a) of the HR emollients of the present invention are oxygen containing emollients, sub groups include alkyl benzoate, alkylene dibenzoate, alkoxyalkyl benzoate or a polyalkylene oxide dibenzoate, or a mixture of two or more sub-classes thereof. The alkyl group often contains at least 10 carbons, in many instances up to 25 carbons. It is often linear, but can alternatively be branched. Especially desirable alkyl groups are found in the range of from 12 to 20 carbons and include dodecyl (lauryl) terdecyl, tetradecyl (myristyl), pentadecyl, hexadecyl (palmityl), octadecyl (stearyl) 2-methyl-heptadecyl (iso-stearyl) and octyldodecyl groups. A mixture of two or more of the alkyl groups can be employed, such as a mixture of C12-C15 alkyl groups. The term alkylated herein includes alkylene groups and the latter are terminated at each end with a benzoate group. The alkylene group often contains from 2 to 6 carbons and can be linear or branched, a suitable example of linear being propylene.

In the alkoxyalkyl benzoates contemplated herein, the alkyl group is terminated by an alkoxy group, which can be monomeric containing for example up to 6 carbons or polymeric such as polyethylene oxide or preferably polypropylene oxide, which conveniently comprises up to 30 units and often from 5 to 20 units. In such compounds, the alkyl group can be selected from the previously identified alkyl groups. Alternatively, the benzoate compound can comprise a polyethylene oxide or polypropylene oxide moiety,

- 15 -

or preferably a block copolymer of ethylene oxide and propylene oxide, terminated at each end by a benzoate group. Mixtures of two or more of the benzoate sub-classes of compounds can be employed. This class a) of HR emollients
5 tends to have a refractive index in the region of 1.465 to 1.49.

Several preferred benzoate compounds which are HR emollients are available from Finetex under their trade name Finsolv.
10 Although Finsolv P is a yellow liquid, its use appears not to result in formulations having a significant hue.

Class b) oxygen-containing HR emollients comprise non-volatile silicone oils. Such compounds commonly comprise
15 alkylphenyl substituted polysiloxanes, and especially methylphenyl polysiloxanes. Desirably, the polysiloxane is short chain and linear, such as a disiloxane, trisiloxane or tetrasiloxane. Particularly desirably, the mole ratio of alkyl (especially methyl) to phenyl substitution is 1:1. It
20 is especially desirable to select within the class of non-volatile polysiloxane materials those which have a viscosity of below 300 centistokes ($300 \times 10^{-6} \text{ M}^2 \text{s}^{-1}$) and advantageously those of below 200 centistokes ($200 \times 10^{-6} \text{ M}^2 \text{s}^{-1}$). In practice, the viscosity of preferred siloxane materials is often in
25 the region of 50 centistokes or higher. The refractive index of preferred non-volatile silicone oils, such as those comprising alkylphenylsiloxanes normally fall within the range of 1.50 to 1.56. Examples of highly preferred non-volatile siloxanes include PDM-7040 and PDM-7050 (trade
30 names) obtainable from Gelest and DC 704 (trade name) obtainable from Dow Corning Inc.

- 16 -

The remaining constituents of the carrier fluid normally comprise other fluids which are miscible with the mixture of emollients or soluble in the final combination, thereby forming an anhydrous fluid carrier. Anhydrous herein
5 indicates that the formulation is free from a distinct aqueous phase, which means in practice that it does not comprise an aqueous emulsion or micro-emulsion. The choice of other carrier fluids is at the discretion of the formulator, within the bounds indicated herein. In
10 practice, they tend to be hydrophobic, although a limited proportion of hydrophilic constituents can be employed, such as those materials which, in the chosen proportions, are still miscible with the remainder of the carrier fluid.

15 For convenience, the oils which can be employed herein as the balance of the fluid carrier are often referred to as LR oil or LR components of the carrier fluid, and these normally have a refractive index of below 1.465. In view of the relative proportions of said hydrocarbon/HR oxygen-
20 containing mixture of emollients and the balance of LR oils in the carrier fluid, the average RI of the carrier is often in the range of from about 1.43 to 1.46, and in many preferred embodiments is at least 1.45. This means that it is not matched to the refractive index of the suspended
25 particulate antiperspirant, so that the observed low scores for visible deposits is normally not achieved by RI matching alone. It is of considerable value to be able to achieve such good results without RI matching, because it is a particularly difficult problem to match constituents
30 sufficiently closely and consistently in commercial scale production and under commercial operating conditions.

- 17 -

The maximum proportion of LR oils in the carrier fluid is the balance above the proportion provided by the HR emollients. The full breadth of the ranges of proportions for LR oils indicated hereinbelow is attainable only to the extent that the balance above the HR emollient proportion permits this.

One suitable class of LR oil comprises volatile liquid silicones, i.e. liquid polyorganosiloxanes. To class as "volatile" such material should have a measurable vapour pressure at 20 or 25°C. Typically the vapour pressure of a volatile silicone lies in a range from 1 or 10 Pa to 2 kPa at 25°C.

It can be desirable for the carrier fluid to include a volatile silicone because it gives a "drier" feel to the applied film after the composition is applied to skin. The carrier fluid often contains from 0 to 50% and particularly from 10 to 40% by weight volatile silicone.

20 Volatile polyorganosiloxanes can be linear or cyclic or mixtures thereof. Preferred cyclic siloxanes include polydimethylsiloxanes and particularly those containing from 3 to 9 silicon atoms and preferably not more than 7 silicon atoms and most preferably from 4 to 6 silicon atoms, otherwise often referred to as cyclomethicones. Preferred linear siloxanes include polydimethylsiloxanes containing from 3 to 9 silicon atoms. The volatile siloxanes normally by themselves exhibit viscosities of below 10^{-5} m²/sec (10 centistokes), and particularly above 10^{-7} m²/sec (0.1 centistokes), the linear siloxanes normally exhibiting a

- 18 -

viscosity of below $5 \times 10^{-6} \text{ m}^2/\text{sec}$ (5 centistokes). The volatile silicones can also comprise branched linear or cyclic siloxanes such as the aforementioned linear or cyclic siloxanes substituted by one or more pendant $-\text{O}-\text{Si}(\text{CH}_3)_3$ groups. Examples of commercially available silicone oils include oils having grade designations 344, 345, 244, 245 and 246 from Dow Corning Corporation; Silicone 7207 and Silicone 7158 from Union Carbide Corporation; and SF1202 from General Electric.

Other LR oils can comprise liquid aliphatic esters. Suitable aliphatic esters usually contain at least one long chain alkyl group, such as esters derived from C_1 to C_{20} alkanols esterified with a C_8 to C_{22} alkanolic acid or C_6 to C_{10} alkanedioic acid. The alkanol and acid moieties or mixtures thereof are preferably selected such that they each have a melting point of below 20°C . These esters include isopropyl myristate, lauryl myristate, isopropyl palmitate, diisopropyl sebacate and diisopropyl adipate. The proportion of aliphatic esters in the carrier fluid is often chosen within the range of 0 to 50% and particularly 0 to 25% of the carrier.

Aliphatic alcohols which are liquid at 20°C may be employed as an LR oil. These include branched chain alcohols of at least 10 carbon atoms e.g. 10 to 25 carbons, such as isostearyl alcohol and octyl dodecanol. The proportion of liquid aliphatic alcohol in the carrier fluid is often chosen within the range of 0 to 50% and particularly 0 to 25% of the carrier.

- 19 -

Other suitable LR oils include aliphatic ethers that are liquid at 20°C, which are derivable from at least one alkanol containing at least 4 carbons and often up to 18 carbons, and which often contain a polyalkylene glycol moiety.

5 Examples of such ethers include myristyl ether derivatives e.g. PPG-3 myristyl ether or lower alkyl ethers of polypropylene glycols such as PPG-14 butyl ether which is commercially available from Amercol under the trade name Fluid AP. The proportion of liquid aliphatic ethers in the
10 carrier fluid is often chosen within the range of 0 to 50% and particularly 0 to 25% of the carrier.

Each of the above-identified classes of LR oils can provide the balance of the carrier fluid. However, it is preferred
15 that at least a fraction of said balance comprises the volatile silicone oil so that the balance generally comprises a mixture of a volatile silicone oil and one of the other LR oils. The weight ratio of volatile silicone oil to other LR oils is often chosen in the range of from
20 1:4 to 3:1.

Structurants

Waxes

25 The term "wax" is conventionally applied to a variety of materials and mixtures which have similar physical properties, namely that:-

they are solid at 30°C and preferably also at 40°C;

they melt to a mobile liquid at a temperature above
30 30°C but generally below 95°C and preferably in a temperature range of 40°C to 90°C;

- 20 -

they are water-insoluble and remain water-immiscible when heated above their melting point.

Waxes are usually hydrocarbons, or silicone polymers, or
5 linear fatty alcohols, esters of fatty acids or glyceride
derivatives or mixtures containing such compounds, possibly
also containing a minority (less than 50%) of other
compounds. Naturally occurring waxes are often mixtures of
compounds which include a substantial proportion, likely to
10 be a majority, of fatty esters.

A wax forms crystals in the carrier fluid when it cools from the heated state during processing.

15 These crystals take various forms including needles and
platelets depending on the individual waxes. Some waxes
form a network of fibrous crystals and can therefore also be
identified as fibre-forming structurants.

20 Examples of hydrocarbon waxes include paraffin wax,
microcrystalline wax and polyethylenes with molecular weight
of 2,000 to 25,000.

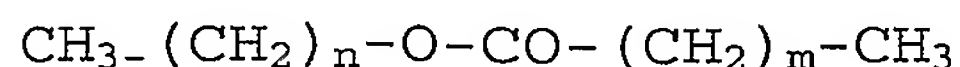
Waxy linear fatty (aliphatic) alcohols normally contain at
25 least 10 and preferably at least 12 carbon atoms, in
practice often not more than 40 carbon atoms and many
preferred alcohols contain from 14 to 25 carbon atoms. Many
formulations which have previously been targeted at low
visible deposits have sought to eliminate altogether or at
30 least severely restrict the proportion of fatty alcohol as
wax structurant therein, indicating a preference for below

- 21 -

1% and more preferably zero per cent. It is to the great advantage of the instant invention, that the benefit of very low visible white deposits can be achieved whilst still permitting the fatty alcohols to be used as a significant contributor to the structuring (solidification) of the carrier fluid.

Two suitable classes of ester waxes include a) glycerol or glycol esters and b) alkyl alkanoate esters. Within class a) the waxes are selected from fatty acid derivatives of glycerol or glycol, such as ethylene glycol. Preferably at least two ester groups are present in the ester waxes. The fatty acid moiety therein normally contains at least 10 carbons and especially from 12 to 24 carbons. Commonly the esters are derived from stearic acid or benhenic acid or a mixture of fatty acids, such as those containing either or both of said acids e.g. C₁₆-C₂₂ fatty acids. It is especially desired to employ glycerol esters. Some esters may be present as components in certain naturally occurring waxes and they these may also be made synthetically. A number of suitable waxes are available from Croda Chemicals under their trade name "Synchrowax", e.g. Synchrowax ERLC.

A second class of ester wax comprises esters which have a melting point of at least 30°C and which satisfy the general formula W1 below:



in which n is from 9 to 39 and m is from 0 to 35.

- 22 -

Within general formula W1, a range of preferred esters comprises those in which n is selected within the range of 14 to 24 and especially 16-22 together with m being selected in the range of 14 to 24 and especially 16 to 22. In second
5 range of preferred esters within the general formula, n is selected in the range of 18 to 38 and m is either 0 or 1. It will be understood that mixtures of esters within each preferred range or mixtures of one preferred range of esters with the other can be employed. Some convenient mixtures
10 include a mixture of a wax comprising esters of n = 14 to 20 and m = 14 to 20 with a wax comprising esters of n = 16 to 20 and m = 14 to 20 or preferably 16 to 20. A number of alkyl alkanoate ester waxes are available from Koster Keunen under their trade name "Kesterwax", e.g. those having
15 designations K62, K69, K80H, K80P, K82H and K82N. Other suitable waxes within this sub-class are available from Strahl and Pitsch under their trade names SP-C36 and SP-C44 (stearyl stearate and behenyl behenate respectively).

20 Mixtures of the ester waxes can be employed, either within either class of ester or a mixture of both classes.

A useful class of waxes comprises those which comprise or consist of glyceride waxes and in particular triglyceride
25 waxes. Many suitable glyceride waxes comprise esters of fatty acids, often containing at least 16 carbon atoms, and especially from 18 to 36 carbon atoms. Examples of suitable ester moieties include stearate, eicosinate and behenate. Certain of them can be derived from naturally occurring oils
30 such as castor oil by hydrogenation. Yet others include tristearin, or are obtainable by hydrogenating vegetable

- 23 -

oils such as rape seed oil. A number of triglyceride waxes are obtainable from Croda Chemicals under their trade name Syncrowax, e.g. grades HRC and HGL-C.

5 Examples of natural waxes or simple derivatives of natural products include castor wax, beeswax, carnauba and candelilla waxes, which are of vegetable origin and mineral waxes from fossil remains other than petroleum. Montan wax, which is an example of mineral wax, includes non-glyceride
10 esters of carboxylic acids, hydrocarbons and other constituents. Other naturally available waxes include spermececi wax, ozokerite, ceresin, baysberry, and synthetic waxes such as Fisher-Tropsch waxes and microcrystalline waxes.

15 Waxes useful in the present invention will generally be those found to thicken water-immiscible oxygen-containing HR emollient oils such as C₁₂₋₁₅ alkyl benzoates and/or non-volatile methylphenylpolysiloxanes, when dissolved therein
20 (by heating and cooling) at a concentration of 5 to 25% by weight.

The wax is normally employed in such an amount that the carrier fluid is structured in combination with any other
25 structurant that is present and the presence of any particulate antiperspirant and other solids. This amount is usually not greater than the weight of the carrier fluid, and in most instances not greater than 30% of the weight of the composition.

30

- 24 -

If a wax is used which forms a network of fibres, the amount of it may commonly be from 4 to 12% by weight of the composition. If a wax is used which does not form such a network, for instance a wax which crystallises as

5 spherulitic needles or as small platelets, the amount is often selected in the range of from 4 to 25% and in many preferred embodiments from 5 to 12% or 10 to 25% of the composition, depending at least in part upon whether the wax is being employed in conjunction with or without a further
10 structurant. Silicone waxes are an example of waxes which crystallise as small platelets.

It is often desirable to employ a combination of waxes.

Preferred combinations include a combination of a glyceride

15 wax with at least one second wax selected from glycerol ester waxes, alkylalkanoate waxes and fatty alcohols, including specifically those waxes mentioned by name hereinbefore. Preferably, the glyceride wax is present in a weight ratio to the second wax or waxes of 1:2 to 1:6 and
20 more preferably from 2:5 to 1:4.

Fibre-forming Gellants

A number of organic compounds are known to possess the

25 ability to gel water-immiscible organic liquids such as water-immiscible hydrocarbon and/or silicone oils. Such materials are generally non-polymeric, i.e. monomers or dimers with molecular weight below 10,000 often below 5,000 or even 1,000 rather than polymers with more than four
30 repeat units or with molecular weight above 10,000.

- 25 -

Gel formation takes place as an exothermic event within a temperature range referred to as the gel point; upon re-heating, melting of the gel takes place as an endothermic event within a temperature range. Such gels can be
5 disrupted by shearing. Although a small partial recovery may then be observed, such gels do not recover their structure for a long time, if at all, unless re-melted.

Materials with this ability to gel water-immiscible organic
10 liquids have been reviewed by Terech and Weiss in "Low Molecular Mass Gelators of Organic Liquids and the Properties of their Gels" Chem. Rev 97, 3133-3159 [1997] and by Terech in Chapter 8, "Low-molecular weight Organogelators" of the book "Specialist surfactants" edited
15 by I D Robb, Blackie Academic Professional, 1997.

It is characteristic of such non-polymeric gellants (structurants), useful in this invention, that:-

they are able to gel the organic liquid in the absence of
20 any disperse phase, when used in sufficient quantity not exceeding 15% by weight;

the structured liquids are obtainable by cooling from an elevated temperature at which the structurant is in solution in the liquid - this hot solution being mobile
25 and pourable;

the (thus obtained) structured liquid becomes more mobile if subjected to shear or stress;

the structure does not spontaneously recover within 24 hours if the sheared liquid is left to stand at ambient
30 laboratory temperature, even though a small partial recovery may be observed;

- 26 -

the structure can be recovered by re-heating to a temperature at which the structurant is in solution in the liquid and allowing it to cool back to ambient laboratory temperature.

5

It appears that such non-polymeric structurants operate by interactions which are permanent unless disrupted by shear or heating. Such structurants form a network of strands or fibres extending throughout the gelled liquid. In some cases these fibres can be observed by electron microscopy, although in other cases the observation of the fibres which are believed to be present is prevented by practical difficulties in preparing a suitable specimen. When observed, the primary fibres in a gel are generally thin (diameter less than 0.5µm, often less than 0.2µm) and appear to have numerous branches or interconnections. Primary fibres may entwine to form a thicker strand.

10 If these fibres are crystalline, they may or may not be the same polymorph as macroscopic crystals obtained by conventional crystallisation from a solvent.

One material which is well known to form such gels is 12-hydroxy stearic acid (12-HSA) which is discussed in Terech et al "Organogels and Aerogels of Racemic and Chiral 12-hydroxy octadecanoic Acid", Langmuir Vol. 10, 3406-3418, 1994. The material is commercially available from Ajinomoto and also from Caschem.

25 30 US-A-5750096 is one of several documents which teaches that gelation can be brought about using esters or amides of 12-

- 27 -

hydroxy stearic acid. The alcohol used to form such an ester or the amine used to form such an amide may contain an aliphatic, cycloaliphatic or aromatic group with up to 22 carbons therein. If the group is aliphatic it preferably
5 contains at least three carbon atoms. A cycloaliphatic group preferably contains at least five carbon atoms and may be a fixed ring system such as adamantyl.

Other fatty acids with C₈ or longer alkyl chains may be used
10 and amides thereof can also be used. A specific example is lauric monoethanolamide also termed MEA lauramide.

N-acyl amino acid amides and esters are employable herein to structure liquids. We have established that they do so by
15 forming fibrous networks. They are described in US patent 3969087. Examples of N-acyl amino acid esters include N α , N δ , dilauroylornithine octyl, decyl, lauryl and stearyl ester, N α , N ϵ , -dilauroyllysine hexyl, octyl, decyl, and lauryl esters, N α , N ϵ , -di(tallowyl) and N α , N ϵ , -
20 di(hydrogenated tallowyl) lysine hexyl, octyl, decyl, and lauryl esters, in which tallowyl indicates the acyl radical of tallow fatty acid.

Examples of N-acylamino acid amides include N-acetyl
25 glutamic acid- α , γ -dilauryl and α , γ -distearyl amides; N-lauroyl glutamic acid diamide, - α , γ -dibutyl, - α , γ -dihexyl, - α , γ -dioctyl, - α , γ -dilauryl and - α , γ -distearyl amides; N-cocoyl glutamic acid, - α , γ -diamide, - α , γ -dibutyl, - α , γ -dihexyl, - α , γ -dioctyl, - α , γ -dilauryl and - α , γ -distearyl
30 amides; N-hydrogenated tallowyl glutamic acid, - α , γ -diamide, - α , γ -dibutyl, - α , γ -dihexyl, - α , γ -dioctyl, - α , γ -

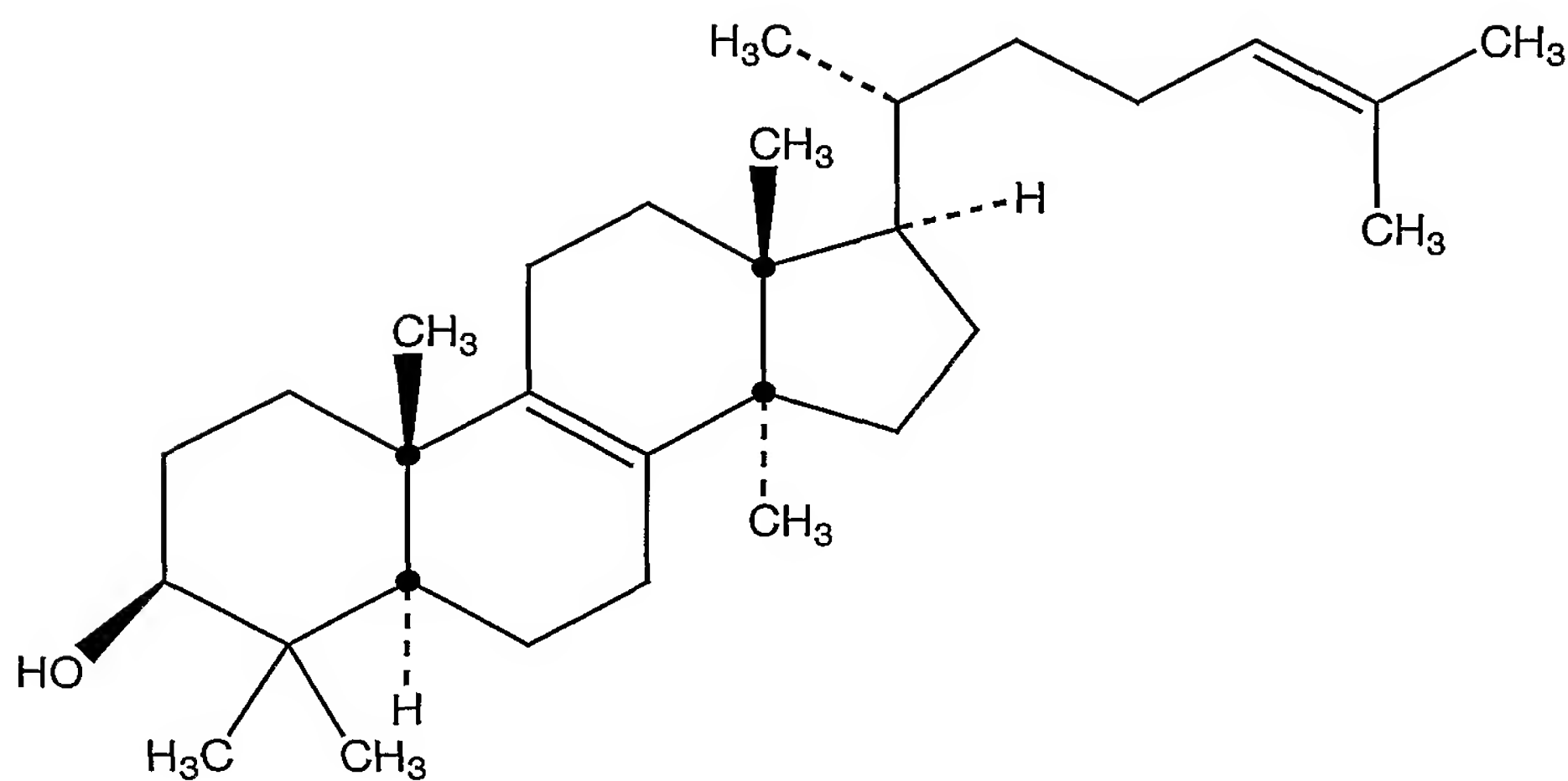
- 28 -

- dilauryl and α,γ -distearyl amides; $N\alpha$, $N\epsilon$, -dicaproyllysine amide, butyl hexyl, octyl, lauryl, and stearyl amides; $N\alpha$, $N\epsilon$, -dicapryloyllysine amide, butyl, dibutyl, hexyl, octyl, lauryl, and stearyl amides; $N\alpha$, $N\epsilon$, -dilauroyllysine amide, butyl, hexyl, octyl, lauryl, and stearyl amides; $N\alpha$, $N\epsilon$, -dicocoyllysine amide, butyl hexyl, octyl, lauryl, and stearyl amides; $N\alpha$, $N\epsilon$, -di(hydrogenated tallowyl) lysine amide, butyl hexyl, octyl, lauryl, and stearyl amides; N-lauroylvaline amide, butyl, hexyl, octyl and lauryl amides; N-lauroyl-phenylalanine amide, butyl, hexyl, octyl and lauryl amides; N-capryloyl leucine amide, butyl, hexyl, octyl and lauryl amides; and N-palmitoylmethionine amide, butyl, hexyl and octyl amides.
- 15 N-Lauroyl-L-glutamic acid di-n-butylamide is particularly favoured and is commercially available from Ajinomoto under their trade designation GP-1.

Further materials which have been disclosed as gelling agents are the amide derivatives of di and tribasic carboxylic acids set forth in WO 98/27954 notably alkyl N,N'dialkyl succinamides.

Lanosterol, as disclosed in US 5635165 mentioned above may suitably be used if the water-immiscible fluid comprises a major fraction of silicone oils. Lanosterol has the following chemical formula.

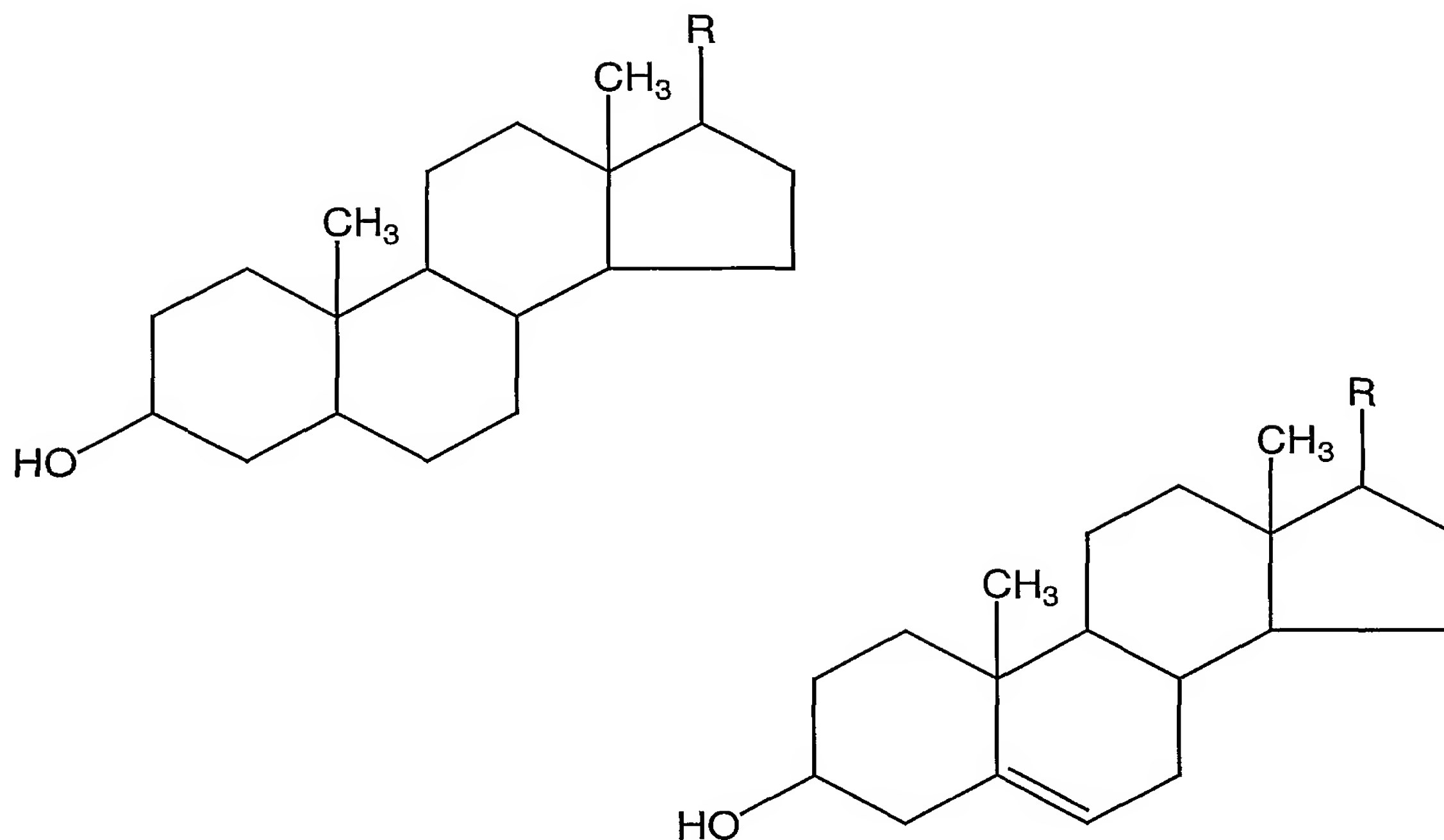
- 29 -



It is commercially available, e.g. from Croda Chemicals Ltd,
and as supplied it contains some dihydrolanosterol. This
5 impurity in the commercial material does not need to be
removed.

A structurant which is the subject of a co-pending
application is a combination of a sterol and a sterol ester.
10 In its preferred form, the sterol satisfies either of the
two formula:-

- 30 -

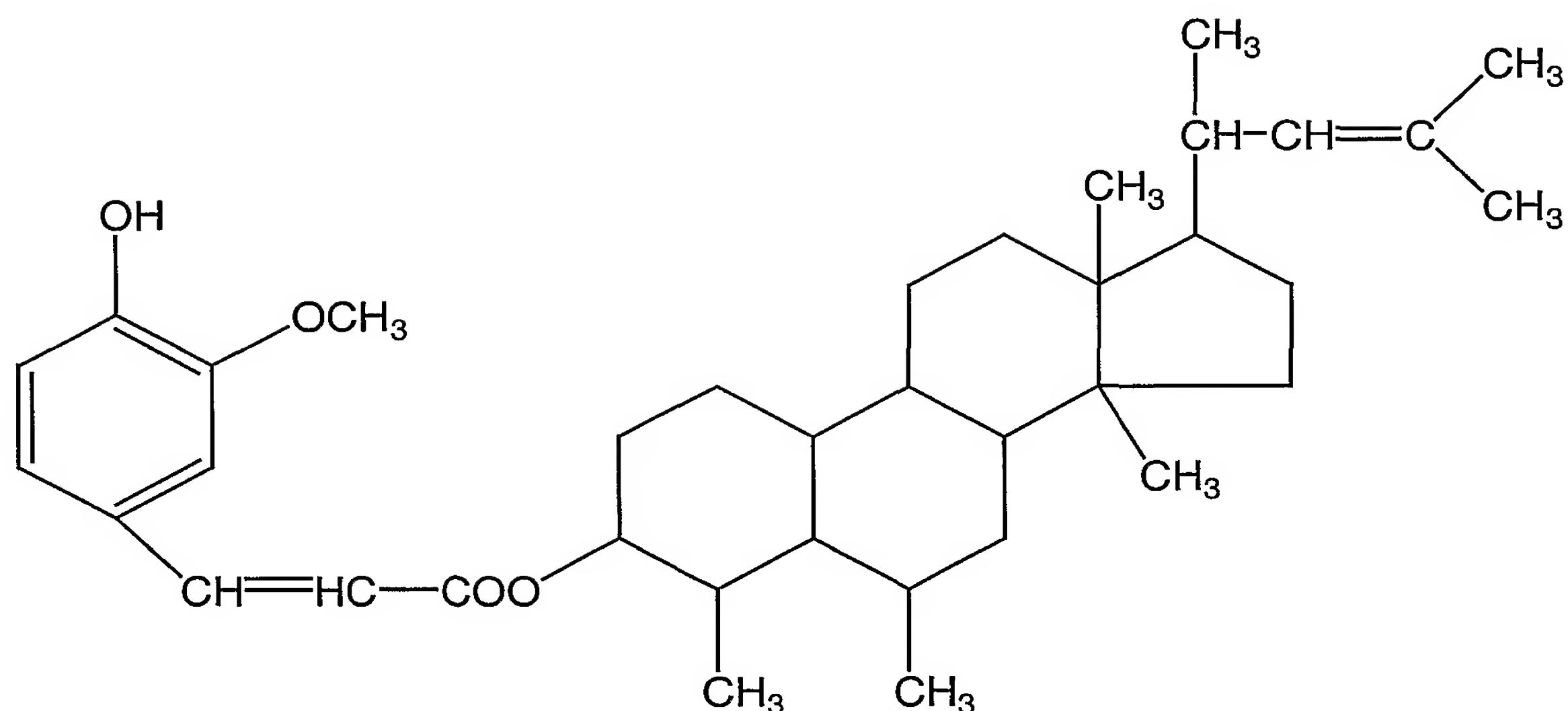


in which R represents an aliphatic, cycloaliphatic or aromatic group, and preferably a linear or branched
 5 aliphatic saturated or unsaturated hydrocarbon group. R desirably contains from 1 to 20 carbons and preferably from 4 to 14 carbons.

It is particularly suitable to employ β -sitosterol or
 10 campesterol or cholesterol, or a hydrogenated derivative thereof, such as dihydrocholesterol, or a mixture of two or more of them. An especially preferred sterol is β -sitosterol.

15 The preferred sterol ester is oryzanol, sometimes referred to as γ -oryzanol which contains material satisfying the following formula:-

- 31 -

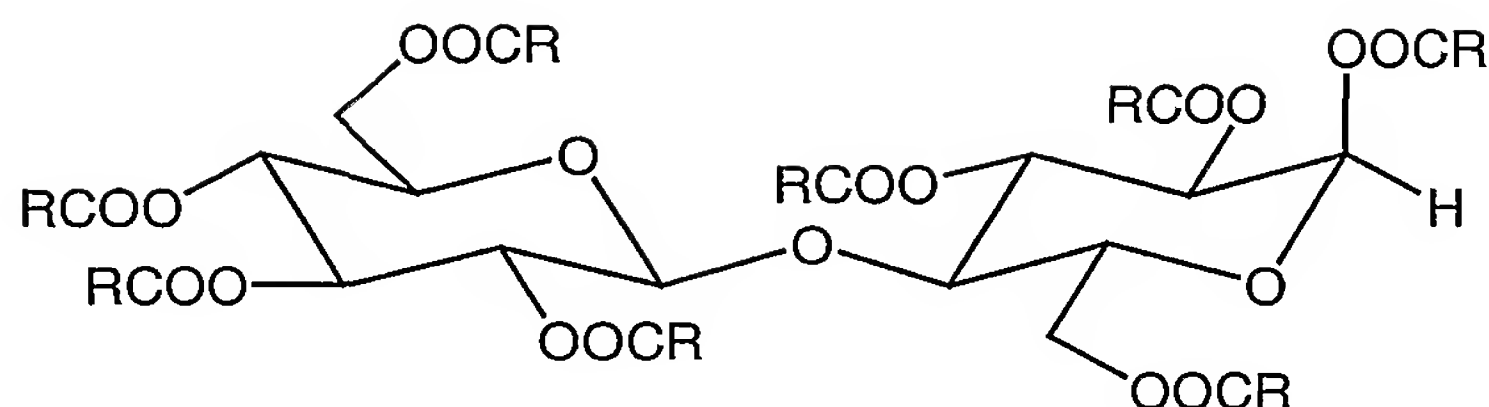


The sterol and sterol ester are used in a mole ratio that is normally selected in the range of from 10:1 to 1:10, especially from 6:1 to 1:4 and preferably in the range of from 3:1 to 1:2. Employment of the two system constituents within such a mole ratio range, and especially within the preferred range facilitates the co-stacking of the constituents and consequently facilitates the formation of a network that is readily able to structure the formulation.

Another structurant which is the subject of a co-pending application and which may be used in this invention is an ester of cellobiose and a fatty acid, preferably of 6 to 13 carbon atoms especially 8 to 10 carbon atoms. Preferably the cellobiose is fully esterified, or nearly so, and is in the α -anomeric form.

The structure of such a compound, in its α -anomeric form is:-

- 32 -



where R is an alkyl or alkenyl chain of 5 to 12 carbon atoms so that the acyl group contains 6 to 13 carbon atoms.

- 5 Particularly preferred acyl groups incorporate a linear alkyl chain of 7 to 9 carbon atoms and are thus octanoyl, nonanoyl or decanoyl.

The acyl groups may have a mixture of chain lengths but it
 10 is preferred that they are similar in size and structure. Thus it is preferred that all of the acyl groups are aliphatic and at least 90% of the acyl groups have a chain length within a range such that the shorter and longer chain lengths in the range differ by no more than two carbon
 15 atoms, i.e. length in a range from $m - 1$ to $m + 1$ carbon atoms where m has a value in a range from 7 to 10.

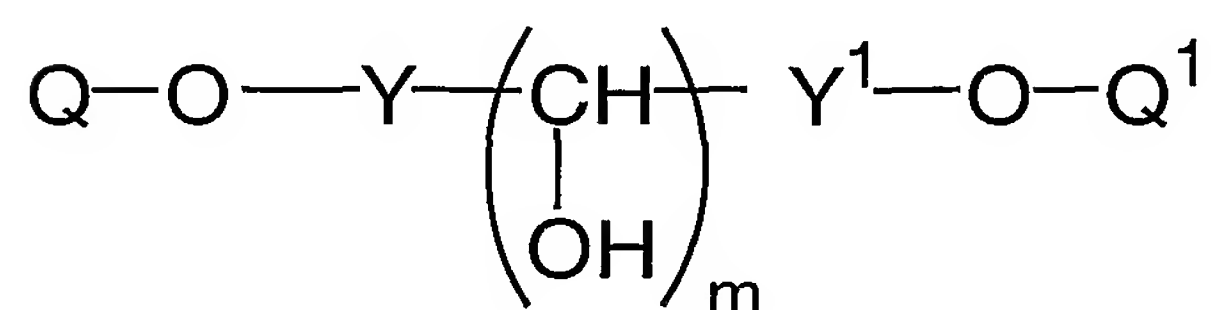
Linear aliphatic acyl groups may be obtained from natural sources, in which case the number of carbon atoms in the
 20 acyl group is likely to be an even number or may be derived synthetically from petroleum as the raw material in which case both odd and even numbered chain lengths are available.

Synthetic methods for the esterification of saccharides are
 25 well known. The esterification of cellobiose has been reported by Takada et al in *Liquid Crystals*, (1995) Volume 19, pages 441-448. This article gives a procedure for the production of the alpha anomers of cellobiose octa-

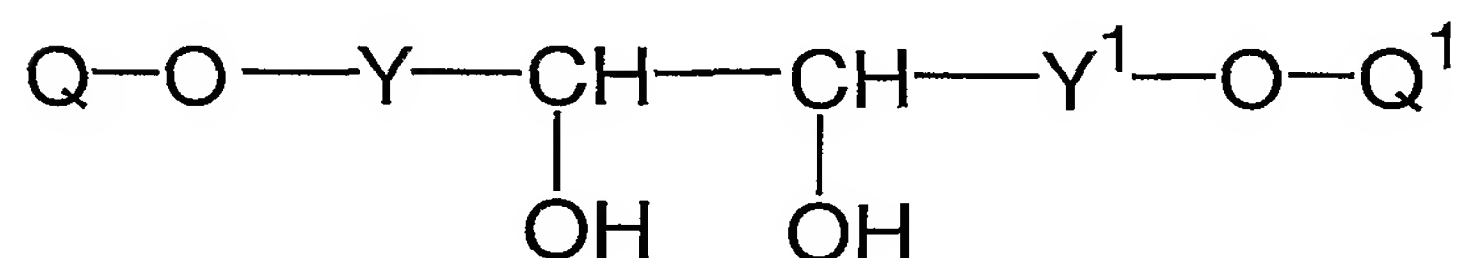
- 33 -

alkanoates by esterification of β -cellobiose using an alkanoic acid together with trifluoroacetic anhydride.

A further example of structurant which is the subject of a
 5 co-pending application is compounds of the following general formula (TI):-



10 It is preferred that m is 2 so that the structurant compounds comply with a general formula (T2):-



15 The groups Y and Y¹ will usually be identical, i.e. both methylene or both carbonyl. The groups Q and Q¹ may not be the same but often will be identical to each other.

If m is 2 and Y and Y¹ are methylene groups, the compound is
 20 a derivative of threitol, which is 1,2,3,4-tetrahydroxybutane, while if m is 2 and Y and Y¹ are carbonyl groups, the compound is a diester of tartartic acid, which is 2,3-dihydroxybutane-1,4-dioic acid.

25 It is preferred that each group Q and Q¹ contains an aromatic nucleus which may be phenyl or, less preferably, some other aromatic group. Thus Q and Q¹ may be groups of the formula

- 34 -

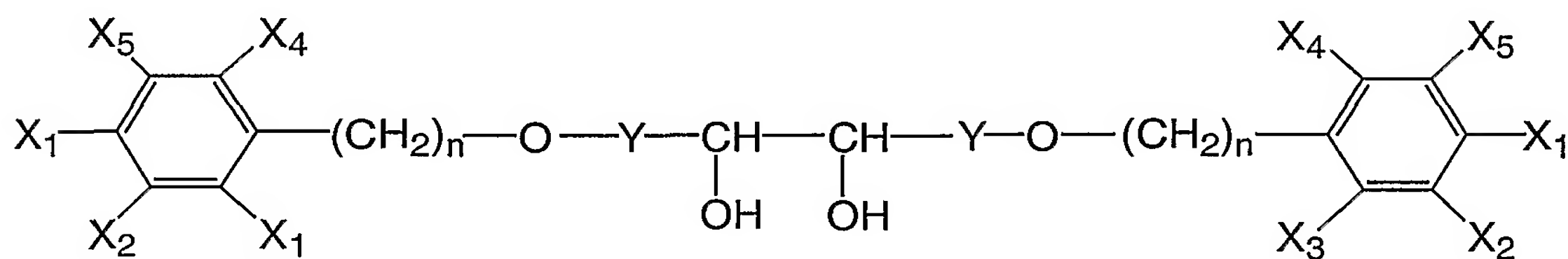


where Ar denotes an aromatic nucleus, notably phenyl or substituted phenyl and n is from 0 to 10.

5

An aromatic nucleus (Ar) is preferably unsubstituted or substituted with one or more substituents selected from alkyl, alkyloxy, hydroxy, halogen or nitro.

10 One substituent may be an alkyl or alkyloxy group with a long alkyl chain. Thus a formula (T3) for preferred structurants of this invention can be given as:-



15

in which

n = 0 to 10, preferably 0 to 3, more preferably 1, 2 or 3;

Y = -CH₂- or >C=O

X₁ = H, Cl, Br, F, OH, NO₂, O-R, or R, where R is an

20 aliphatic hydrocarbon chain with 1 to 18 carbon atoms.

X₂ to X₅ are each independently H, Cl, Br, F, OH, NO₂, OCH₃, or CH₃

In these formulae above, the central carbon atoms which bear
25 hydroxy groups are chiral centres. Thus if m = 2,

Y and Y¹ are the same and Q and Q¹ are the same, the compounds will exist as R,R and S,S optically active forms as well as an optically inactive R,S form.

- 35 -

These compounds may be used as their optically active R,R or S,S forms or as a mixture of the two - which may be a racemic mixture.

5 Compounds within the general formula (TI) above are available commercially. Also, syntheses of these compounds have been given in scientific literature where the compounds were being used as intermediates for purposes not related to the present invention. Thus syntheses of threitol
10 derivatives can be found in:

Kataky et al, J. Chem Soc Perkin Trans vol. 2 page 321
[1990] Tamoto et al, Tetrahedron Vol. 40 page 4617 [1984],
and Curtis et al, J.C.S. Perkin I Vol. 15 page 1756 [1977].

15

Preparations of tartrate esters are found at:

Hu et al J. Am. Chem. Soc. Vol. 118, 4550 [1996] and
Bishop et al J. Org Chem Vol 56 5079 [1991].

20 One structurant which it is eminently desirable to employ in conjunction with a further structurant, be it wax or non-polymeric fibre forming structurant is 12-hydroxystearic acid (abbreviated herein to 12-HSA). It is especially desirable to employ a combination of 12-HSA with either an
25 N-acyl amino acid amide, or with an alkyl alkanoate ester wax. The weight ratio of 12-HSA to the N-acyl amino acid amide is often selected in the weight ratio range of from 1:1 to 5:1. In such combinations, the weight of the 12-HSA is often chosen within the range of 3 to 8% by weight and
30 that of the N-acyl amino acid amide in the range of from 1 to 5% by weight. The weight ratio of 12-HSA to the

- 36 -

selected wax is often in the range of 1:1 to 1:3. The weight of 12-HSA in such a combination is often selected in the range of from 3 to 8% by weight and that of the wax from 4 to 12% by weight.

5

Antiperspirant Actives

10 An essential constituent of an antiperspirant formulation is an antiperspirant active. Antiperspirant actives are preferably incorporated in an amount of from 0.5-60%, particularly from 5 to 30% or 40% and especially from 5 or 10% to 30 or 35% of the weight of the composition.

15 Antiperspirant actives for use herein are often selected from astringent active salts, including in particular aluminium, zirconium and mixed aluminium/zirconium salts, including both inorganic salts, salts with organic anions and complexes. Preferred astringent salts include aluminium,
20 zirconium and aluminium/zirconium halides and halohydrate salts, such as chlorohydrates.

Aluminium halohydrates are usually defined by the general formula $Al_2(OH)_xQ_y \cdot wH_2O$ in which Q represents chlorine,
25 bromine or iodine, x is variable from 2 to 5 and $x + y = 6$ while wH_2O represents a variable amount of hydration.

Especially effective aluminium halohydrate salts, known as activated aluminium chlorohydrates, are described in EP-A-6739 (Unilever NV et al), the contents of which
30 specification is incorporated herein by reference. Some activated salts do not retain their enhanced activity in the

- 37 -

presence of water but are useful in substantially anhydrous formulations, i.e. formulations which do not contain a distinct aqueous phase.

5 Zirconium actives can usually be represented by the empirical general formula: $\text{ZrO}(\text{OH})_{2n-nz}\text{B}_z \cdot w\text{H}_2\text{O}$ in which z is a variable in the range of from 0.9 to 2.0 so that the value $2n-nz$ is zero or positive, n is the valency of B , and B is selected from the group consisting of chloride, other
10 halide, sulphamate, sulphate and mixtures thereof. Possible hydration to a variable extent is represented by $w\text{H}_2\text{O}$. Preferable is that B represents chloride and the variable z lies in the range from 1.5 to 1.87. In practice, such zirconium salts are usually not employed by themselves, but
15 as a component of a combined aluminium and zirconium-based antiperspirant.

The above aluminium and zirconium salts may have co-ordinated and/or bound water in various quantities and/or
20 may be present as polymeric species, mixtures or complexes. In particular, zirconium hydroxy salts often represent a range of salts having various amounts of the hydroxy group. Zirconium aluminium chlorohydrate may be particularly preferred.

25 Antiperspirant complexes based on the above-mentioned astringent aluminium and/or zirconium salts can be employed. The complex often employs a compound with a carboxylate group, and advantageously this is an amino acid. Examples
30 of suitable amino acids include dl-tryptophan, dl- β -

- 38 -

phenylalanine, dl-valine, dl-methionine and β -alanine, and preferably glycine which has the formula $\text{CH}_3\text{CH}(\text{NH}_2)\text{CO}_2\text{H}$.

It is highly desirable to employ complexes of a combination
5 of aluminium halohydrates and zirconium chlorohydrates together with amino acids such as glycine, which are disclosed in US-A-3792068 (Luedders et al). Certain of those Al/Zr complexes are commonly called ZAG in the literature. ZAG actives generally contain aluminium,
10 zirconium and chloride with an Al/Zr ratio in a range from 2 to 10, especially 2 to 6, an Al/Cl ratio from 2.1 to 0.9 and a variable amount of glycine. Actives of this preferred type are available from Westwood, from Summit and from Reheis.

15 Other actives which may be utilised include astringent titanium salts, for example those described in GB 2299506A.

The proportion of solid antiperspirant salt in a composition
20 normally includes the weight of any water of hydration and any complexing agent that may also be present in the solid active.

In the present invention, the composition takes the form of
25 a suspension in which antiperspirant active in particulate form is suspended in the water-immiscible liquid carrier. Such a composition does not have any separate aqueous phase present and may conveniently be referred to as "substantially anhydrous" although it should be understood
30 that some water may be present bound to the antiperspirant active or as a small amount of solute within the water-

- 39 -

immiscible liquid phase. In such compositions, the particle size of the antiperspirant salts often falls within the range of 0.1 to 200µm with a mean particle size often from 3 to 20µm. Both larger and smaller mean particle sizes can
5 also be contemplated such as from 20 to 50µm or 0.1 to 3µm.

Optional ingredients

10 In addition to the ingredients listed above, the anhydrous, topically-effective compositions of the present invention also can include other optional ingredients that are conventionally included in topically applied cosmetic compositions. Optional ingredients in compositions of this
15 invention can include disinfectants, for example at a concentration of up to about 10% w/w. Suitable deodorant actives can comprise deodorant effective concentrations of antiperspirant metal salts, deoperfumes, and/or microbicides, including particularly bactericides, such as
20 chlorinated aromatics, including biguanide derivatives, of which materials known as triclosan (Irgasan DP300 TM), chlorhexidine and Tricloban TM, warrant specific mention. A yet another class comprises biguanide salts such as available under the trade mark Cosmosil TM.

25

At the option of the manufacturer, and save as excluded hereinbefore, inclusion of a polymeric structurant can be contemplated. Polymeric structurants which can be employed can comprise organo polysiloxane elastomers such as reaction
30 products of a vinyl terminated polysiloxane and a cross linking agent or alkyl or alkyl polyoxyalkylene-terminated

- 40 -

poly(methyl substituted) or poly(phenyl substituted)
siloxanes. Suitable elastomers have been disclosed in for
example WO 98/00097 and WO 98/18438. A number of polyamides
have also been disclosed as structurants for water-
5 immiscible liquids.

It is desirable to include within the formulation one or
more wash-off aids, often in a proportion of up to about 10%
by weight, especially up to about 5% by weight and
10 particularly from 0.5 to 3% by weight based on the
formulation.

Such wash off aids commonly comprise nonionic surfactants
and especially nonionic surfactants which contain a
15 polyalkylene oxide moiety, the residue of a fatty acid or
fatty alcohol and optionally the residue of an aliphatic
polyhydric alcohol linking group. Although, the surfactants
may comprise a single fatty residue, they preferably contain
two residues. Preferably, the surfactant is an ester
20 surfactant, and especially a diester surfactant. The
polyalkylene oxide is often polyethylene oxide, or
polypropylene oxide or mixed polyethylene oxide/propylene
oxide, the polymer containing from 3 to 50 and especially
from 5 to 20 alkylene oxide units. The fatty acid or
25 alcohol often contains from 12 to 24 carbons, and in many
instances is linear, examples including 16, 18 or 22 linear
carbons. Especially preferred wash-off aids herein comprise
polyethylene oxide diesters of fatty alcohols containing 16
to 22 linear carbons, such as PEG-8 distearate.

- 41 -

One important class of optional constituents comprises fragrances. They can be incorporated into the anhydrous, topically-effective compositions in an amount of from 0% to about 5% and often from 0.2 to 1.5% by weight based on the
5 total weight of the composition. Fragrance-containing compositions of the present invention, when applied to skin, fix a substantive fragrance film on the skin that resists moisture, but that can be removed by washing.

10 One other class of ingredients comprises moisturising agents such as humectants. These include propylene glycol, sorbitol and especially glycerol. Moisturising agents often comprise from 0 to 5% by weight of the formulation, and if employed, it is desirable that the amount is chosen such
15 that the agent is retained within the carrier fluid.

Yet other optional ingredients that can be included in the anhydrous composition of the present invention include, but are not limited to, drying agents, like talc or DRY FLO
20 (aluminium starch octenylsuccinate); preservatives; and dyes. Generally, such optional ingredients are present in a composition of the present invention in an amount of about 10% or less by weight. In addition, an organoclay can be included in a composition of the present invention as an
25 additional suspending agent in an amount of up to 20% by weight of the composition. An organoclay is potentially helpful as an anti-caking agent to maintain a particulate topically-effective compound homogeneously dispersed throughout the composition. An exemplary organoclay is a
30 quaternised three-layer clay exfoliated with a polar solvent, like a quaternised montmorillonite clay exfoliated

- 42 -

with propylene carbonate. Such clays are available under the trade name "Bentone". Other inorganic materials which can be incorporated, for example as a thickener for the formulation, comprise particulate silica, such as fumed
5 silica, suitably in an amount of up to 5%.

Still other cosmetic adjuncts can include:-

skin feel improvers, such as talc (mentioned hereinabove) or
10 finely divided polyethylene or glyceryl fatty esters, e.g. glyceryl stearate, incorporated, for example, in an amount of up to about 10% w/w;

skin benefit agents such as allantoin or lipids, for example in an amount of up to 5% w/w; and

15 skin cooling agents, such as menthol and menthol derivatives, often in an amount of up to 2% w/w.

Many of the stick formulations produced in accordance with the present invention are opaque. However, it is possible
20 to obtain sticks having an appearance which is at least slightly translucent, for example by so selecting the constituents of the carrier fluid and the nature of the antiperspirant active that the refractive index of the carrier fluid and dispersed solid differs by no more than
25 about 0.06, preferably employing antiperspirant active particulates which lack substantial internal voids.

Substantially internal void-free actives can be obtained by milling void-containing actives.

30 Whilst the ingredients disclosed herein are effective for preparing the antiperspirant formulations of the instant

- 43 -

invention, prospective manufacturers should keep abreast of advances in scientific understanding of their effect on humans and particularly on skin before selling the resultant compositions for topical application to skin.

5

The compositions described herein can be produced by conventional processes for making suspension solid sticks.

10 A convenient process sequence for suspension antiperspirant formulations comprises first mixing the structurant or mixture of structurants, namely the wax(es), the non-polymeric fibre-forming gellant(s) or a mixture of both with the carrier at a temperature that is high enough to melt the structurant. Thereafter, particulate
15 antiperspirant active can be blended with the carrier solution and the blend is formed into a solid mass by cooling, for example by being introduced into its dispensing container at a temperature that is often 5 to 10°C above its normal setting temperature. The process normally includes a
20 suitable filling process, such as a pour fill process (sometimes gravity -fed injection) or injection at elevated pressure into a dispensing container such as a barrel where it is cooled or allowed to cool to ambient.

25 The compositions herein are suitable for applying topically to human skin, and particularly antiperspirant compositions to axillae, thereby reducing observable perspiration.

30

- 44 -

Product Packages

The compositions of this invention are structured liquids which are firm in appearance. A composition of this invention will usually be marketed as a product comprising a container with a quantity of the composition therein, where the container has an aperture for the delivery of composition, and means for urging the composition in the container towards the delivery aperture. Conventional containers take the form of a barrel of oval cross section with the delivery aperture(s) at one end of the barrel. An alternative cross section is round.

A composition of this invention is typically sufficiently rigid that it is not apparently deformable by hand pressure. It is suitable for use as a stick product in which a quantity of the composition in the form of a stick is accommodated within a container barrel having an open end at which an end portion of the stick of composition is exposed for use. The opposite end of the barrel is closed.

Generally the container will include a cap to cover its open end and a component part which is sometimes referred to as an elevator or piston fitting within the barrel and capable of relative axial movement along it. In such packaging, the stick of composition is accommodated in the barrel between the piston and the open end of the barrel. The piston is used to urge the stick of composition along the barrel. The piston and stick of composition may be moved axially along the barrel by manual pressure on the underside of the piston using a finger or rod inserted within the barrel. Another

- 45 -

possibility is that a rod attached to the piston projects through a slot or slots in the barrel and is used to move the piston and stick. Preferably the container also includes a transport mechanism for moving the piston. One
5 transport mechanism comprises a threaded rod which extends axially into the stick through a correspondingly threaded aperture in the piston, and means mounted on the barrel for rotating the rod. Conveniently, the rod is rotated by means of a hand-wheel mounted on the barrel at its closed end,
10 i.e. the opposite end to the delivery opening.

The component parts of such containers are often made from thermoplastic materials, for example polypropylene or polyethylene.

15

Having described the invention in general terms, specific embodiments thereof will now be described in some detail by way of example only.

- 20 List of Ingredients (trade name, INCI name, supplier)
- 1) DC245, cyclomethicone (Dow Corning Inc) [RI = 1.3997]
 - 2) Stearyl alcohol (Henkel/Cognis)
 - 3) SP-C44 Ester Wax, Behenyl Behenate, (Strahl and Pitsch)
 - 4) Castorwax MP80, hydrogenated castor oil (CasChem)
 - 25 5) Kessco PEG 400 DS - PEG-8 Distearate (Stepan Company)
 - 6) Polyiso 275 - polyisobutene 275 (Fancol) [RI = 1.440]
 - 7) Finsolv TN , C12-15 Alkyl Benzoate (Finetex) [RI = 1.4841]
 - 8) Silkflo 364 NF, polydecene (Albemarle) [RI = 1.4544]
 - 30 9) Talc 1626, talc (Suzorite)

- 46 -

- 10) Reach 908, Al/Zr tetrachlorohydrex glycine complex
(Reheis)
- 11) Synchrowax ERLC, C₁₈₋₃₆ glycol esters (Croda)
- 12) AZAG 7167, Al/Zr tetrachlorohydrex glycine complex
5 (Summit)
- 13) Mineral Oil, Sirius M70 (Dalton) [RI= 1.4625]
- 14) Kesterwax K82H, C₂₀₋₄₀ alkyl stearate (Koster Keunen)
- 15) Kesterwax K62, C₁₆₋₂₂ alkyl stearate behenate (Koster
Keunen)
- 10 16) Glyceryl stearate, Cutina MD (Henkel)
- 17) Fumed Silica, Aerosil 200 (Degussa)
- 18) Finsolv TPP, mixture of C₁₂₋₁₅ Alkyl Benzoate /
dipropylene glycol dibenzoate / PPG-15 stearyl ether
benzoate (Finetex) [RI = 1.4915]

15

General Preparative Method

The preparations in Example 1 were made by the general
20 preparative route.

The structurant or structurants were weighed into a round-
bottomed flask or a beaker. The amounts of carrier oil(s)
were then weighed into the flask, and the mixture was
25 stirred with an overhead stirrer and heated using a
isomantle or hotplate until all of the structurant had
dissolved. The source of heat was then removed and the
mixture left to cool to about 85°C. While still stirring the
weighed amount of the particulate antiperspirant active, and
30 where appropriate other particulate materials, including
talc, was or were then added with stirring into the hot

- 47 -

5 mixture. The mixture was permitted to cool until its
temperature had reached about 10°C above the solidification
temperature of the formulation, judged either a trial run or
on the basis of experience with other formulations using the
same structurant(s), temperature sensitive ingredients such
as the perfume were added using a Pasteur pipette. The
mixture continued to be stirred and cool and was poured into
stick barrels at approximately 5°C above its normal
solidification temperature, i.e. solidification at 1
10 atmosphere pressure and without being subjected to treatment
which can alter the solidification temperature. The sticks
were then left to cool in the laboratory and solidify.
Characterisation was carried out when the sticks had been
stored at room temperature for at least 24 hours.

15

Example 1 - Wax Structured Opaque Sticks

The formulations made in Example 1 and their properties are
summarised in Table 1 below.

- 48 -

Table 1

	Ex 1.1	Ex 1.2	Ex 1.3	Ex 1.4	Ex 1.5	Ex 1.6
DC245 (1)	9.75	12.25	9.35	11.25		
Stearyl alcohol (2)	17.0	18.0	20.0	17.0		
Behenyl behenate (3)		5.0				
Castorwax (4)	3.5	3.0	5.0	5.0	4.5	4.5
PEG-8 Distearate (5)	1.0	1.0	1.0	1.0		
Polyisobutene (6)	22.0	18.0	20.0	20.0		
Finsolv TN (7)	22.0	18.0	20.0	20.0	28.5	45.6
Polydecene (8)						11.4
Talc 1626 (9)	2.0	2.0	2.0	2.0		
Reach 908 (10)	22.0	22.0	22.0	22.0		
Syncrowax (11)					13.5	13.5
AZAG 7167 (12)					24.0	24.0
Mineral Oil (13)					28.5	
Fragrance Oil	0.75	0.75	0.65	0.75	1.0	1.0

- 49 -

Example 2

Further sticks were made in accordance with the general preparative method of Example 1 and with the formulation summarised in Table 2 below, in which n/d = not carried out.

Table 2

	Ex2.1	Ex2.2	Ex2.3	Ex2.4
	% by weight			
K82H (14)	10.4	10.4	10.4	
K62 (15)	5.6	5.6	5.6	
Stearyl alcohol (2)	2	2	2	
Castorwax (4)				5
Behenyl Behenate (3)				14
AZAG 908 (10)	24	24	24	24
Finsolv TN (7)	25	23		28.5
Finsolv TPP (18)			30	
Polyisobutene (6)	10	10	10	5
Glyceryl Stearate (16)	5	5		
PEG-8 Distearate (5)		2		2
DC245 (1)	17	17	17	17
talc (9)				2
silica (17)				1.7
Perfume	1	1	1	0.8
	Properties			
Penetrometer Hardness (mm)	7.6	n/d	n/d	9.5
Whiteness on Black Wool (24hrs)	22.1	19.8	29.1	24.3
Whiteness on grey Wet or Dry paper (24hrs)	31.4	31.3	31.4	32.5

- 50 -

Measurement of Properties

i) Hardness of a stick using a Penetrometer

The hardness and rigidity of a composition which is a firm
5 solid can be determined by penetrometry.

A suitable procedure is to utilise a lab plant PNT
penetrometer equipped with a Seta wax needle (weight 2.5
grams) which has a cone angle at the point of the needle
10 specified to be $9^{\circ}10' \pm 15'$. A sample of the composition
with a flat upper surface is used. The needle is lowered
onto the surface of the composition and then a penetration
hardness measurement is conducted by allowing the needle
with its holder to drop under a total weight, (i.e. the
15 combined weight of needle and holder) of 50 grams for a
period of five seconds after which the depth of penetration
is noted. Desirably the test is carried out at a number of
points on each sample and the results are averaged.
Utilising a test of this nature, an appropriate hardness for
20 use in an open-ended dispensing container is a penetration
of less than 30 mm in this test, for example in a range from
2 mm to 30 mm. Preferably the penetration is in a range
from 5 mm to 20 mm.

25 In a specific protocol for this test measurements on a stick
were performed in the stick barrel. The stick was wound up
to project from the open end of the barrel, and then cut off
to leave a flat, uniform surface. The needle was carefully
lowered to the stick surface, and then a penetration
30 hardness measurement was conducted. This process was
carried out at six different points on the stick surface.

- 51 -

The hardness reading quoted is the average value of the 6 measurements.

ii) Deposition and whiteness of deposit

5

Deposition

Another test of the properties of a composition is the amount of the composition which is delivered onto a surface
10 when the composition is drawn across that surface
(representing the application of a stick product to human skin). To carry out this test of deposition, a sample of the composition with standardised shape and size is fitted to apparatus which draws the sample across a test surface
15 under standardised conditions. The amount transferred to the surface is determined as an increase in the weight of the substrate to which it is applied. If desired the colour, opacity or clarity of the deposit may subsequently be determined.

20

A specific procedure for such tests used apparatus to apply a deposit from a stick onto a substrate under standardised conditions and then measures the mean level of white deposits using image analysis.

25

The substrates used normally are:

a: 12 x 28cm strip of black Worsted wool fabric.

b: 12 x 28cm strip of grey abrasive paper (3M™ P800 WetorDry™ Carborundum paper)

30

- 52 -

The substrates are weighed before use. The sticks are previously unused and with domed top surface unaltered.

5 The apparatus comprises a flat base to which a flat substrate is attached by a clip at each end. A pillar having a mounting to receive a standard size stick barrel is mounted on an arm that is moveable horizontally across the substrate by means of a pneumatic piston.

10 Each stick is kept at ambient laboratory temperature overnight before the measurement was made. The stick is advanced to project a measured amount from the barrel. The barrel is then placed in the apparatus and a spring positioned to biased the stick against the substrate with a
15 standardised force. The apparatus is operated to pass the stick laterally across the substrate eight times. The substrate is carefully removed from the rig and re-weighed.

Whiteness of Deposit

20

The deposits from the previous test can be assessed for their whiteness after an interval of 24 hours approximately.

This can be done using a Sony XC77 monochrome video camera
25 with a Cosmocar 16mm focal length lens positioned vertically above a black table illuminated from a high angle using fluorescent tubes to remove shadowing. The apparatus is initially calibrated using a reference grey card, after the fluorescent tubes has been turned on for long enough to give
30 a steady light output. A cloth or Carborundum paper with a deposit thereon from the previous test is placed on the

- 53 -

table and the camera used to capture an image. An area of the image of the deposit is selected and analysed using a Kontron IBAS image analyser. This notionally divides the image into a large array of pixels and measured the grey
5 level of each pixel on a scale of 0 (black) to 255 (white). The average of the grey intensity is calculated. This is a measure of the whiteness of the deposit, with higher numbers indicating a whiter deposit. It is assumed that low numbers show a clear deposit allowing the substrate colour to be
10 seen.

Refractive Index

The refractive index of a liquid is measured using a
15 standard refractometer at 22°C.

The refractive index of a particulate solid is measured at 22°C using a comparative method in which the particulate material is suspended in a range of fluids of known
20 refractive index, and the mixture which gave the highest clarity to the eye of a skilled person is taken to be the refractive index of the particulate solid.

- 54 -

Claims

1. An anhydrous antiperspirant formulation comprising a particulate antiperspirant suspended in a hydrophobic carrier fluid which is structured by an effective amount of a structurant, characterised in that at least 45% by weight of said carrier fluid comprises a mixture of at least two emollient oils comprising a hydrocarbon oil and an oxygen-containing emollient oil, preferably in a weight ratio of from 7:3 to 1:9, said mixture having a refractive index of at least 1.46 and said structurant comprises a wax or a non-polymeric fibre-forming gellant.
2. An antiperspirant stick according to claim 1 characterised in that the mixture of emollient oils of higher RI has a refractive index of at least 1.465.
3. An antiperspirant stick according to claim 1 or 2 characterised in that the carrier fluid comprises at least 60% by weight of said mixture.
4. An antiperspirant stick according to claim 3 characterised in that said mixture comprises from 70 to 99% by weight of the carrier fluid.
5. An antiperspirant stick according to any preceding claim characterised in that the hydrocarbon oil and the oxygen-containing emollient oil are present in the mixture in a weight ratio of from 3:2 to 1:5.

- 55 -

6. An antiperspirant stick according to claim 5 characterised in that the hydrocarbon oil and the oxygen-containing emollient oil are present in the mixture in a weight ratio of from 55:45 to 45:55 or from about 30:70 to about 17:83.
7. An antiperspirant stick according to any preceding claim characterised in that the carrier fluid is free from an alkoxy cinnamate.
8. An antiperspirant stick according to any preceding claim characterised in that the hydrocarbon oil is selected from mineral oils, polydecene, hydrogenated polyisobutenes, and α -olefins.
9. An antiperspirant stick according to any of claims 1 to 3 characterised in that the oxygen-containing emollient oil comprises an alkyl benzoate, an alkylene dibenzoate, an alkoxyated alkyl benzoate or a polyalkylene oxide dibenzoate, or a mixture of two or more thereof.
10. An antiperspirant stick according to claim 7 characterised in that oxygen-containing emollient oil comprises an alkyl benzoate in which the alkyl group contains from 12 to 20 carbon atoms, or mixture of two or more of said alkyl benzoate compounds.
11. An antiperspirant stick according to claim 8 characterised in that the alkyl group in the alkyl benzoate is selected from octyldodecyl, isostearyl,

- 56 -

dodecyl to pentadecyl, and mixtures of dodecyl to pentadecyl.

13. An antiperspirant stick according to any preceding
5 claim characterised in that the stick contains said wax
as structurant in an amount of from 10 to 25% by weight
of the stick.
14. An antiperspirant stick according to any preceding
10 claim characterised in that the stick contains as
structurant one or more waxes selected from fatty
alcohols, glyceride waxes, glycol ester waxes, and
alkylalkanoate waxes.
- 15 15. An antiperspirant stick according to claim 14
characterised in that it contains said non-polymeric
fibre-forming gellant as structurant in an amount of
from 4 to 12% by weight of the stick.
- 20 16. A process for making an antiperspirant stick comprising
the steps of:-
i. incorporating into a liquid carrier a structurant
at an effective concentration and in an amount
sufficient to structure the carrier to render it solid
25 at 20°C
ii. rendering the structurant-containing mixture or
one or more of its constituents mobile at an elevated
temperature
iii. mixing the liquid carrier with an antiperspirant
30 active to form an antiperspirant-containing mixture,

- 57 -

steps 2 and 3 being conducted either before, after or simultaneously with step 1

iv. introducing the mobile mixture into moulding means and

5 v. cooling or permitting the mobile mixture to cool to a temperature at which it is structured, characterised in that at least 45% by weight of said carrier fluid comprises a mixture of at least two emollient oils comprising a hydrocarbon oil and an
10 oxygen-containing emollient oil, preferably in a weight ratio of from 7:3 to 1:9, said mixture having a refractive index of at least 1.46 and said structurant comprises a wax or a non-polymeric fibre-forming gellant.

15

17. A cosmetic method of reducing or controlling axillary sweating comprising applying topically to skin an anhydrous antiperspirant formulation comprising a particulate antiperspirant suspended in a water-
20 immiscible carrier fluid which is structured by an effective amount of a structurant, characterised in that at least 45% by weight of said carrier fluid comprises a mixture of at least two emollient oils comprising a hydrocarbon oil and an oxygen-containing
25 emollient oil, preferably in a weight ratio of from 7:3 to 1:9, said mixture having a refractive index of at least 1.46 and said structurant comprises a wax or a non-polymeric fibre-forming gellant.

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- 58 -

18. An antiperspirant product comprising an antiperspirant formulation in the form of a stick disposed within a packaging which enables a portion of the stick to be exposed for topical application to skin characterised in that the formulation comprises a particulate antiperspirant suspended in a water-immiscible carrier fluid which is structured by an effective amount of a structurant, characterised in that at least 45% by weight of said carrier fluid comprises a mixture of at least two emollient oils comprising a hydrocarbon oil and an oxygen-containing emollient oil, preferably in a weight ratio of from 7:3 to 1:9, said mixture having a refractive index of at least 1.46 and said structurant comprises a wax or a non-polymeric fibre-forming gellant.

INTERNATIONAL SEARCH REPORT

 Inter... I Application No
 PCT/GB 01/02882

A. CLASSIFICATION OF SUBJECT MATTER
 IPC 7 A61K7/32

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 972 319 A (LINN ELIZABETH ET AL) 26 October 1999 (1999-10-26) column 2, line 55 -column 3, line 10; claims 1,2,7-9,11-13 ----	1,3-7,9, 13-18
X	WO 91 04009 A (GILLETTE CO) 4 April 1991 (1991-04-04) page 2, paragraph 2 page 4, paragraph 1; claims 1-3,6,7,9-15,22 ----	1,3,5, 7-15,17, 18
A	US 4 293 544 A (ELMI STEELE J) 6 October 1981 (1981-10-06) example II ----- -/-	1-18

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

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- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

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- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

6 December 2001

Date of mailing of the international search report

12/12/2001

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INTERNATIONAL SEARCH REPORT

Intern al Application No
PCT/GB 01/02882

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>WO 97 16163 A (PROCTER & GAMBLE) 9 May 1997 (1997-05-09) the whole document -----</p>	1-18

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Claims Nos.: 1-18 (partially)

Present claims 1-18 relate to a product comprising "a mixture of at least two emollient oils comprising a hydrocarbon oil and an oxygen-containing emollient oil", wherein said mixture is defined by reference to the following parameter " having a refractive index of at least 1.46 ".

The use of this parameter in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameters the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Consequently, the search has been restricted to products comprising a mixture of at least two emollient oils comprising a hydrocarbon oil and an oxygen-containing emollient oil.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 01/02882

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